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Dislocations and twins in nanocrystalline Ni after severe plastic deformation: the effects of grain size

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Abstract

Deformation microstructures have been investigated in nanocrystalline (nc) Ni with grain sizes in the 50–100 nm range. It was found that deformation twinning started to occur in grains of ∼90 nm, and its propensity increased with decreasing grain size. In most of the nc grains dislocations were observed as well, in the form of individual dislocations and dipoles. It is concluded that dislocation-mediated plasticity dominates for grain sizes in the upper half, *i.e.* 50–100 nm, of the nanocrystalline regime.

Keywords: Nanocrystalline metal; Plastic deformation; Twinning; Dislocation; Ni

1. Introduction

The mechanisms of plastic deformation in nanocrystalline (nc) metals, with grain sizes typically smaller than 100 nm, have been a subject of considerable research in recent years [1–6]. Molecular dynamics (MD) simulations [2–4] have shown the presence of size effects: (i) grain boundary (GB) sliding at very fine grain sizes (e.g. 3–10 nm), (ii) partial dislocation-mediated processes (PDMPs) at several tens of nanometers (e.g. 20–50 nm), and (iii) dislocation-mediated plasticity near the upper limit of nanocrystalline regime (*i.e.*, 50–100 nm). The PDMPs form the basis of three mechanisms of plastic deformation, *i.e.* deformation twinning, formation of extended stacking faults, and full dislocations [2–4]. On the experimental side, the PDMPs, in particular for deformation twinning, have been verified in nc Al [1,7], Cu [8], Pd [9], and Ni [10–12] by transmission electron microscopy (TEM) studies.

It is noted, however, that all the PDMPs observed in nc metals such as Ni were mostly for grain sizes in the range of 20–50 nm. There remain, therefore, questions regarding the dependence of the onset of the PDMPs such as deformation twinning on grain size. Up to date, there is no report of the dependence of PDMPs on grain sizes in the same nc metal (e.g., Ni) under the same deformation conditions.

The objective of this work is to study the deformation microstructure in nc Ni with grain size in the range of 50–100 nm, to explore at which grain size the PDMPs begin to operate, extending the grain size range covered before using electroplated nc Ni [10–14]. Our examinations will also verify if full dislocations indeed control the plasticity in this grain size regime, as predicted by MD simulations.

2. Experimental

A high-purity polycrystalline Ni (purity: 99.998 wt.%) was used in this study. The average grain size of the starting material was determined to be ∼40 μm. The nc Ni was produced by the technique of surface mechanical attrition treatment (SMAT) [15]. The SMAT process was performed under vacuum for 50 min with a 50 Hz vibrating frequency at room temperature. Using this technique, it is possible to capture the deformation microstructure in various grain size regimes, by examining the treated surface layer at different depths. Following SMAT, the deformed microstructure was observed in high-resolution TEM (HRTEM). The thin foils for HRTEM observation were prepared by means of cutting, grinding, dimpling and a finally ion milling or twin-jet polishing at low temperature.

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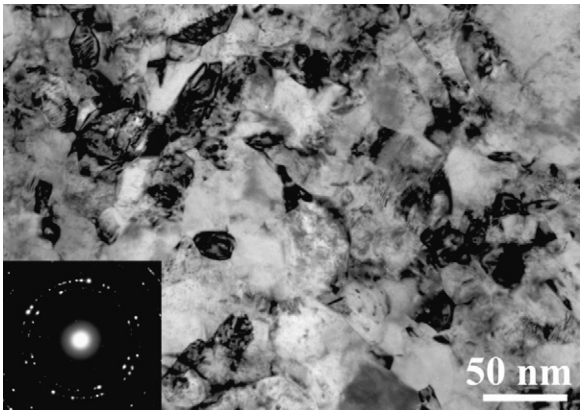

Fig. 1. TEM image showing the nc grains of Ni. The diffraction pattern is shown as inset.

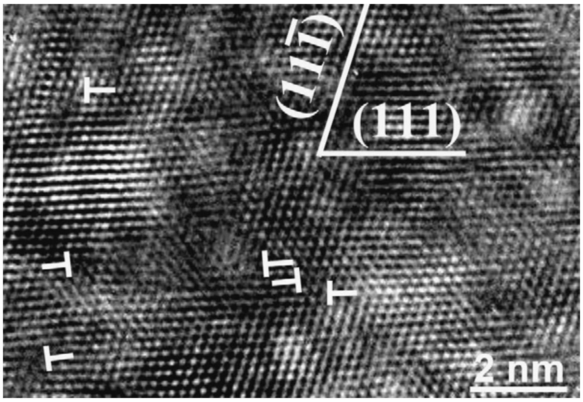

Fig. 3. HRTEM image showing dislocations (indicated by their extra-half planes) of a nc grain of 80 nm.

3. Results

HRTEM observations reveal that the nc grains, with grain sizes smaller than 100 nm, are formed in the deformed layer from the top surface to ∼30 μm deep after SMAT, whereas the submicron-sized grains (100–500 nm) are formed in the deeper layer (∼30–70 μm). Fig. 1 is a TEM image showing the deformation microstructure of Ni at a depth of ∼25 μm from the free surface. Most grains are equiaxed with a mean grain size of ∼65 nm. The inset shows the electron diffraction pattern.

We first made extensive observations for the submicron-sized grains (∼50 in number with grain sizes in the range 100–500 nm). No deformation twinning was observed. The onset of deformation twinning was observed to occur with the reduction of grain size to below 100 nm. In our study, the largest grain having deformation twins was ∼93 nm in size. Fig. 2 is a TEM image showing deformation twinning occurring in a grain of ∼80 nm. The arrows indicate the twin boundaries. Clearly, the regions on two sides of the grain have been transformed into twins, together with a twin plate going across the whole grain. It is likely that these twins were heterogeneously nucleated at the GBs of the grain and grew *via* the emission of Shockley partial dislocations from GBs [4,7–11]. The inset in Fig. 2 shows a HRTEM image of the framed area enclosing a twin boundary.

It is further observed that deformation twinning occurs only in 20% of the grains observed (6 out of ∼30 grains with sizes in the 50–100 nm range). In contrast, dislocation-mediated plasticity still dominates in these larger nc grains. Fig. 3 is a HRTEM image showing dislocations in an 80 nm grain. They are present in the form of individual dislocation as well as dipoles. The dislocation density in the area of Fig. 3 is $\sim 1.9 \times 10^{17}\ \mathrm{m}^{-2}$.

It is noteworthy that the grain size strongly affects the twinning propensity: twins were observed in 50% of the smaller nc grains (20–50 nm), as we reported before [10,11], but only in 20% of the larger nc grains (50–100 nm). Fig. 4 gives an example showing a 20 nm nc grain with a twin plate marked by lines. A Fourier-transformed diffraction pattern indicating the twin relationship is shown as inset.

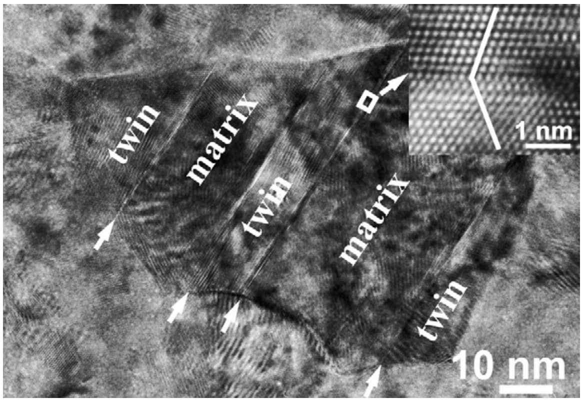

Fig. 2. TEM image showing deformation twins in a nc grain. The twin boundaries are indicated by arrows. The inset shows a HRTEM image of the framed area enclosing a twin boundary.

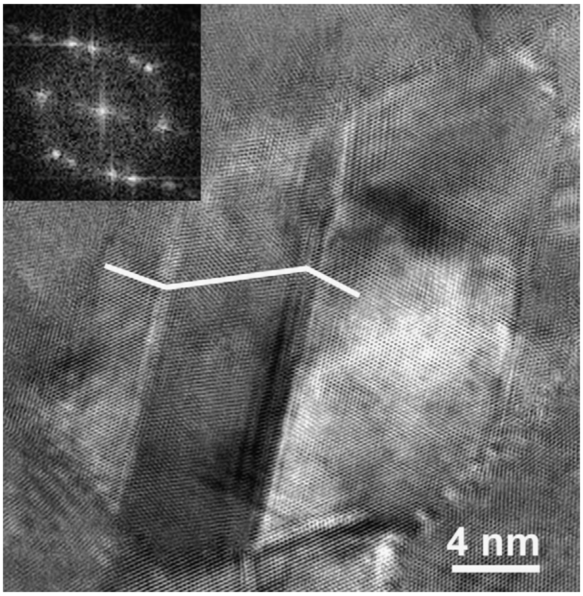

Fig. 4. HRTEM image showing a deformation twin in a nc grain. The diffraction pattern of the twin is shown as inset.

4. Discussion and concluding remarks

Conventional dislocation sources such as the Frank-Read source may be expected to operate in these larger nc grains (50–100 nm). Our post mortem observations shown in Fig. 3 have indeed revealed dislocation debris in such grains. Statistically, most of the larger nc grains (50–100 nm) experienced plastic deformation-mediated by dislocation slip, rather than deformation twinning. This corroborates the expectation that dislocation-mediated plasticity may still dominate in the upper nanocrystalline regime. In MD simulations [2], all three PDMPs and multiple twinning events are seen for nc Ni deformed under high stresses. In our experiments, severe plastic deformation does provide high stresses, especially when the grain size enters the nc regime. There will also be build-up of local stress concentrations in the deforming nanostructure. At sufficiently high stress levels, twinning becomes a possible deformation response. This apparently happened for grain sizes around 90 nm in our case. The twinning propensity increases with decreasing grain size.

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